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IS 4750 (1996): Sorbitol Powder, Food Grade [FAD 8: Food Additives]

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सोरबिटॉल पाउडर, खाद्य ग्रेड — विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

SORBITOL POWDER, FOOD GRADE — SPECIFICATION

(First Revision)

First Reprint August 2001

ICS 67.220.20

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

September 1996 **Price Group 3**

AMENDMENT NO. 1 MARCH 2005
TO
IS 4750 : 1996 SORBITOL POWDER, FOOD GRADE — SPECIFICATION

(First Revision)

[*Page* 2, *Table* 1, *Sl No.* (i), *col* 2] — Substitute 'Purity, as *d*-sorbitol $C_6H_{14}O_6$, percent by mass, on dry basis, *Min* ' for the existing.

[*Page* 2, *Table* 1, *Sl No.* (i), *col* 3] — Substitute '91' *for* '91 or 99 1)' and delete the footnote.

(*Page* 2, *Table* 1) — Insert the following at the end of the table:

Sl No.	Characteristic	Requirement	Methods of Test, Ref to	
			Cl No. of Annex A	Other Standards
(1)	(2)	(3)	(4)	(5)
xii)	Lead (as Pb), mg/kg, *Max*	1	—	Cl 15 of IS 1699 : 1995

(*Page* 4, *Annex* A, *clause* **A-1.2**) — Delete the clause and the text contained therein along with the sub-clauses.

(FAD 8)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 2 JUNE 2008
TO
IS 4750 : 1996 SORBITOL POWDER, FOOD GRADE — SPECIFICATION

(First Revision)

(*Page* 3, *Annex* A, *clause* **A-1.0**) — Delete the clause and the text contained therein.

(*Page* 3, *Annex* A, *clause* **A-1.1**) — Substitute '**Column Chromatographic Method**' for the existing.

(FAD 8)

Reprography Unit, BIS, New Delhi, India

Food Additives Sectional Committee, FAD 8

FOREWORD

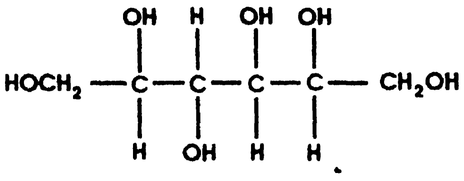
This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council

With the increased production of processed foods, manufacturers have started adding a large number of substances, generally in small quantities, to improve the appearance, flavour, texture or storage properties of the processed foods As certain impurities in these substances could be harmful, it is necessary to have a strict quality control of these food additives A series of standards was, therefore, prepared by this Institution to cover purity and identification of these substances These standards would help in checking purity, which requires to be checked at the stage of manufacture, for it is extremely difficult (and in many cases impossible) to detect the impurity once these substances have been added to the processed foods Besides, these standards are intended to guide the indigenous manufacturers in making their product conform to specifications that are accepted by scientists, health authorities and international bodies, and the consumer industries to use them within the quantity permitted by the health authorities

Sorbitol powder food grade used as a food additive is permitted under '*Prevention of Food Adulteration Rules* 1955 These rules, *inter-alia* prescribe

> The listed food additives permitted for use in certain foods shall be sold only under the BIS Certification Mark Sorbitol, food grade, is one among the listed additives

Chemical Names and Formula — The recognized chemical names are *d*-sorbitol, *d*-glucitol, *d*-sorbite, *d* sorbol, and 1,2,3,4 5 6-hexanehexol Empirical formula is $C_6H_{14}O_6$ Its molecular weight is 182 17 Structural formula is



$$\begin{array}{ccccccccccc} & & OH & & H & & OH & & OH & & \\ & & | & & | & & | & & | & & \\ HOCH_2 & — & C & — & C & — & C & — & C & — & CH_2OH \\ & & | & & | & & | & & | & & \\ & & H & & OH & & H & & H & & \end{array}$$

This standard was first published in 1968 and is being revised to make the following additions/changes

a) To upgrade the standard by reducing the moisture content considerably and specifying the limit for nickel
b) To provide a separate clause for description including the solubility property to keep it in line with the Food Chemicals Codex NRC
c) To substitute the requirement of lead by heavy metals and the corresponding change in method of test
d) To replace the requirements of non-reducing sugar by total sugar

This standard contains Item (i) in Table 1 which calls for an agreement between the purchaser and the supplier

In the formulation of this standard, considerable amount of assistance has been derived from 'Food chemical codex issued by the National Academy of Sciences, National Research Council, Washington

The Bureau has already published IS 3987 1966 'Specification for sorbitol solution (70 percent) for use in cosmetic preparations'

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 1960 Rules for rounding off numerical values (*revised*)' The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard

Indian Standard
SORBITOL POWDER, FOOD GRADE — SPECIFICATION
(First Revision)

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and tests for sorbitol powder, food grade.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard

IS No.	*Title*
266 1993	Specification for sulphuric acid (*third revision*)
1070 1992	Reagent grade water (*third revision*)
1699 1995	Methods of sampling and test for synthetic food colours (*second revision*)
4818.1996	Sorbic acid, food grade (*first revision*)
5058.1996	Sodium citrate, food grade (*first revision*)

3 DESCRIPTION

3.1 Sorbitol is white hygroscropic powder having a sweet taste Its solution in water is clear, colourless, odourless, syrupy liquid having a pleasant sweet taste Sorbitol is very soluble in water and slightly soluble in ethanol.

NOTE The solubility is intended only as information regarding approximate solubility and is not to be considered as a quality requirement and is of minor significance as a means of identification or determination of purity, and dependence must be placed on other specifications

4 REQUIREMENTS

4.1 Identification

Dissolve about 5 g of the material in 6 ml of water, add 7 ml of methanol, 1 ml of benzaldehyde, and 1 ml of hydrochloric acid, and shake in a mechanical shaker until crystals appear. Filter with aid of suction, dissolve the crystals in 20 ml of boiling water containing 1 g of sodium carbonate, filter while hot, cool the filtrate, filter with suction, wash with 5 ml of methanol-water mixture (1 in 2), and dry in air The sorbitol monobenzylidene so obtained shall melt between 173°C and 177°C when determined by the method given in Annex A of IS 4818:1996

4.2 The material shall also conform to the requirements given in Table 1

5 PACKING AND MARKING

5.1 Packing

5.1.1 The material shall be securely packed in containers with minimum access to air. The containers shall be such as to preclude contamination of the contents with metals or other impurities.

5.2 Marking

5.2.1 Each container shall be legibly and indelibly marked with the following information·

a) Name of the material, including the words 'Food Grade';
b) Name and address of the manufacturer,
c) Net mass when packed;
d) Batch or code number,
e) Expiry date; and
f) Any other requirements as specified under the *Standards of Weights and Measures (Packaged Commodities) Rules*, 1977/ *Prevention of Food Adulteration Rules*, 1955

5.2.2 *BIS Certification Marking*

The product may also be marked with the Standard Mark.

5.2.2.1 The use of the Standard mark is governed by the provisions of *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standrds

6 SAMPLING

6.1 Representative samples of the material shall be drawn according to the method prescribed in **4** of IS 1699·1995.

7 QUALITY OF REAGENTS

7.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070.1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the experimental results

Table 1 Requirements for Sorbitol Powder, Food Grade

(*Clause* 4.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Cl No of Annex A	Other Standards
(1)	(2)	(3)	(4)	(5)
i)	Purity, as *d* sorbitol $C_6H_{14}O_6$, percent by mass, *Min*	91 or 99[1]	A-1	–
ii)	Moisture, percent by mass, *Max*	1.0	A-2	--
iii)	Melting range			
	a) Metastable	92.5 °C to 93.5 °C	—	Annex A of IS 4818 : 1996
	b) Stable	96.0 °C to 97.5 °C		
iv)	Reducing sugars, percent by mass, *Max*	0.2	A-3	—
v)	Sulphated ash, percent by mass, *Max*	0.1	A-4	--
vi)	Sulphates (as SO_4), percent by mass, *Max*	0.01	A-5	—
vii)	Chlorides (as Cl), percent by mass, *Max*	0.005	A-6	—
viii)	Arsenic (as As), mg/kg, *Max*	3	-	15 of IS 1699 : 1995
ix)	Heavy metals (as Pb), mg/kg	10	—	Annex E of IS 5058 : 1996
x)	Total sugar, percent by mass as dextrose, *Max*	1	A-7	-
xi)	Nickel, mg/kg, *Max*	2	A-8	–

[1] Depending on the method of test used for analysis

ANNEX A

(*Table* 1)

ANALYSIS OF SORBITOL

A-1 DETERMINATION OF *d*-SORBITOL

A-1.0 Two methods have been specified, one using column chromatogram other without it. Either method could be used as agreed to between the purchaser and the supplier [*see* Table 1, Item (i)]. Purity shall be minimum 91 percent by the method I and 99 percent by the Method II.

A-1.1 Method I — Column Chromatographic Method

A-1.1.1 *Principle of Method*

Sorbitol is first isolated chromatographically and then oxidized with a known excess of potassium periodate. The excess of potassium periodate is determined by titration iodometrically.

A-1.1.2 *Apparatus*

A-1.1.2.1 *Chromatographic column*

Place a pledge of slightly tapered chromatographic tube, 38 mm × 230 mm, which is inserted in a 500-ml suction flask. Add with full vacuum 80 g of a mixture of 5 parts of very fine chromatographic fullers earth and 1 part of chromatographic siliceous earth in portions of 2 to 3 g, tapping the tube gently after each addition. Apply suction gently to compress the adsorbent, add an additional 10 g of adsorbent to the column to a height of about 200 mm. Level the top with a spatula and cover it with a disc of filter paper.

A-1.1.3 *Reagents*

A-1.1.3.1 *Isopropyl alcohol solvent*

Prepared by mixing 60 ml of water and 340 ml of isopropyl alcohol.

A-1.1.3.2 *Alkaline potassium permanganate solution*

Dissolve 10 g of sodium hydroxide and 1 g of potassium permanganate in 100 ml of water.

A-1.1.3.3 *Potassium periodate reagent*

Prepared by mixing 40 ml of dilute sulphuric acid (1 in 20) and 60 ml of potassium periodate solution (1 in 1 000).

A-1.1.3.4 *Potassium iodide*

A-1.1.3.5 *Standard sodium thiosulphate solution* — 0.02 N

A-1.1.3.6 *Starch solution*

A-1.1.3.7 *Sulphuric acid*

A-1.1.4 *Procedure*

A-1.1.4.1 To about 450 mg of the material, accurately weighed, add 1 ml of water and 5 ml of *isopropyl* alcohol solvent, mix and pour the solution on the prepared chromatographic column. Complete the addition with the aid of three 10 ml portions of the solvent waiting after each addition until the solvent has practically disappeared on the column. Attach a separating funnel containing 325 ml of the solvent to the tube by means of a rubber stopper inserted to a point 16 mm above the top of the column. Admit enough solvent to make a 1-cm layer and adjust the separating funnel so that its tip is just below the solvent surface. As soon as an even front of the liquid has advanced about 1.3 to 2.0 cm down the tube apply full suction and open the separator stopcock to admit solvent so that the effluent collects initially at a rate of approximately 3 ml per minute and continues until all of the solvent has been added taking precaution to avoid letting the column run day.

A-1.1.4.2 When all of the solvent has been added from the separator and the liquid layer just disappears into the column, continue full vacuum for 1 to 3 minutes, then disconnect the system promptly and extrude the column on to a glazed paper. Paint several streaks length of the column with alkaline potassium permanganate solution. Note and mark the zones where the streaks are decolourized. Cut out the second zone, which usually starts about 45 mm to 50 mm from the top of the column and extends 125 mm to 150 mm ,with a sharp blade. Return this portion of the column to the tube in which a fresh pledget of cotton has been placed on a porous plate. Replace the separating funnel and, with the aid of gentle suction, draw the column down the chromatographic tube snugly on the cotton. Elute the adsorbed sorbitol with 200 ml of water applying full vacuum when water has advanced about 13 mm down the column. Transfer the eluate to a 500-ml volumetric flask make up to volume with water and mix.

A-1.1.4.3 Pipette 5 ml of the dilute eluate into a 250 ml conical flask. Add 50 ml of potassium periodate reagent which has been acidified with 5 drops of sulphuric acid. Heat the solution on a steam bath for 15 minutes and cool to room temperature. Add 1 g of potassium iodide, allow to stand for 5 minutes and titrate with sodium thiosulphate solution using starch indicator near the end point. Perform a blank using water in place of the material.

A-1.1.5 *Calculation*

$$d\text{ Sorbitol, percent by mass} = \frac{100\,(B - S) \times 1.822 \times N}{W}$$

where

B = volume in ml of standard sodium thiosulphate solution used in the blank titration,

S = volume in ml of standard sodium thiosulphate solution used in the material titration,

N = normality of standard sodium thiosulphate solution and

W = mass in g of the material taken for the test.

A-1.2 Method — II

A-1.2.1 *Reagents*

A-1.2.1.1 *Sodium periodate solution* — 0.1 N

A-1.2.1.2 *Potassium hydrogen carbonate*

A-1.2.1.3 *Sodium arsenite solution*

A-1.2.1.4 *Potassium iodide*

A-1.2.1.5 *Iodine solution* — 0.1 N

A-1.2.2 *Procedure*

Weigh accurately about 50 mg of sample and mix with 40 ml of sodium periodate solution and allow to stand for one hour. Then add 1 g of potassium hydrogen carbonate, 50 ml of sodium arsenite solution and about 2 g of potassium iodide. Titrate against iodine solution using starch as indicator. Carry out a blank. The difference between the two titrations corresponds to the volume of sodium periodate taken up by the sorbitol powder. Each ml of 0.1 N sodium periodate corresponds to 0.001 821 8 g of sorbitol.

A-2 DETERMINATION OF MOISTURE

A-2.1 Apparatus

A-2.1.1 *Oven* — maintained at 80 ± 1°C and pressure 5 mm of mercury.

A-2.1.2 *Weighing Bottle* — glass-stoppered, shallow.

A-2.2 Procedure

A-2.2.1 Weigh accurately about 2 g of the powdered sample in the tared weighing bottle. Distribute the sample as evenly as practicable to a depth of about 5 mm. Place the bottle containing the sample (uncovered) in the oven maintained at 80 ± 1°C and 5 mm pressure of mercury. Remove the bottle from the oven after six hours, close the bottle promptly and allow it to come to room temperature in a desiccator, weigh it.

A-2.2.2 Calculate the loss on drying percent by weight.

A-3 TEST FOR REDUCING SUGARS

A-3.1 Principle of Method

Reducing sugars are determined through treatment with Fehling's solution, the reduced copper oxide is weighed.

A-3.2 Reagent

A-3.2.1 *Alkaline Cupric Tartrate Solution (Fehling's Solution)*

a) *Copper solution* (A) — Dissolve 36.44 g of carefully selected small crystals of cupric sulphate showing no trace of efflorescence or adhering moisture, in sufficient water and make it to 500 ml. Store this solution in small tight containers.

b) *Alkaline tartrate solution* (B) — Dissolve 173 g of crystallized potassium sodium tartrate and 50 g of sodium hydroxide in sufficient water and make it to 500 ml. Store this solution in small alkali-resistant containers.

Mix exactly equal volumes of solutions A and B at the time of conducting the test.

A-3.2.2 *Diethyl Ether*

A-3.2.3 *Ethyl Alcohol* — 95 percent (*v/v*)

A-3.3 Procedure

Transfer 7 g of the material, accurately weighed, to a 500 ml beaker with the aid of 35 ml of water and mix. Add 50 ml of alkaline cupric tartrate solution. Cover the beaker with a watchglass and heat the mixture at such a rate that it comes to boil in approximately 4 minutes. Boil for exactly 2 minutes. At once add 100 ml of cold, recently boiled water and immediately collect the precipitated cuprous oxide in a tared gooch crucible which has been previously washed with hot water, alcohol and ether and dried at 105°C for 30 minutes. Thoroughly wash the collected cuprous oxide on the filter with hot water, then with 10 ml of alcohol and finally with 10 ml of ether. Dry at 105°C for 30 minutes.

A-3.3.1 The material shall be taken to have satisfied the requirement in Table 1 if the weight of cuprous oxide does not exceed 50 mg.

A-4 DETERMINATION OF SULPHATED ASH

A-4.1 Reagent

A-4.1.1 *Concentrated Sulphuric Acid* — (*See* IS 266 : 1993)

A-4.2 Procedure

Weigh accurately about 2 g of the material in a tared crucible. Ignite, gently at first, until the material is thoroughly charred, cool, moisten the residue with 1 ml of sulphuric acid and ignite gently till the carbon is completely consumed. Cool the crucible in a desiccator and weigh.

NOTE — Carry out the ignition in a place protected from air currents and use as low a temperature as possible to effect the combustion of carbon.

A-4.3 Calculation

$$\text{Sulphated ash, percent by mass} = \frac{W_1}{W_2} \times 100$$

where

W_1=mass in g of the residue, and

W_2=mass in g of the material taken for the test.

A-5 SULPHATE TEST

A-5.1 Standard Sulphate Solution

Dissolve 148 mg of anhydrous sodium sulphate in water and dilute to 100 mm. Transfer 10 ml of this solution to a 1 000 ml volumetric flask, dilute to volume with water, and mix. Each ml of the final solution contains 10 mg of sulphate (SO_4).

A-5.2 Procedure

Dissolve 2 g of the test sample in 30 to 40 ml of water, neutralize to litmus external indicator with dilute hydrochloric acid, if necessary. Then add 1 ml of dilute hydrochloric acid. To the clear solution or filtrate add 3 ml of torium chloride, dilute to 50 ml with water, and mix. Allow it to stand for 10 minutes. Compare the turbidity, if any, with that produced in a control solution containing 20 ml (equivalent to 200 mg) of sulphate solution and the same quantities of the reagents used for the sample.

Any turbidity produced by the sample shall not exceed the turbidity shown in the control solution.

A-6 CHLORIDE TEST

A-6.1 Standard Chloride Solution

Dissolve 165 mg of sodium chloride in water and dilute to 100 ml. Transfer 10 ml of this solution to a 1 000 ml volumetric flask, dilute to volume with water, and mix. Each ml of the final solution contains 10 mg of chloride.

A-6.2 Procedure

Dissolve 400 g of the test sample in 30 to 40 ml of water, neutralize to litmus external indicator with nitric acid, if necessary, then add 1 ml of silver nitrate, dilute to 50 ml with water, and mix. allow to stand for 5 minutes protecting from direct sunlight. Compare the turbidity, if any, with that produced in a control solution containing 2 ml of the standard chloride solution (equivalent to 20 mg) and the reagents in the same quantities which are used for the sample.

The turbidity produced, if any, by the sample shall not exceed that produced by the control solution.

A-7 TOTAL SUGAR

A-7.1 Procedure

A-7.2 Transfer 2.1 g of material into a 250-ml flask fitted with a ground-glass joint, add 40 ml of approximately 0.1 N hydrochloric acid, attach a reflux condenser, and reflux for 4 h. Transfer the solution to a 400 ml beaker, rinsing the flask with about 10 ml of water, neutralize with 6 N sodium hydroxide, and continue as directed under reducing sugars, beginning with 'Add 50 ml of alkaline cupric tartrate' till the weight of the cuprous oxide does not exceed 50 mg.

A-8 DETERMINATION OF NICKEL

A-8.1 Procedure

A-8.2 Dissolve 10 g of the sample in sufficient water to produce 20 ml, add 3 ml of bromine water and 2 ml of a 20 percent of mass/volume solution of citric acid, mix and add 10 ml of 6 N ammonia and 1 ml of a 1 percent *m/v* solution of dimethylglyoxime in alcohol. Mix, dilute to 50 ml with water and allow to stand for 5 minutes; any colour produced is not more intense than that produced by similarly treating, 1 ml of standard nickel solution (equivalent to 2 ppm) in place of sample.

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